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(54) Title: PROCESS

(57) Abstract: A process for the preparation of a vanadyl sulphate solution with a specified molar concentration from a first starting material containing vanadium pentoxide (V_2O_5) and a second starting material containing vanadium trioxide (V_2O_3). The first and second starting materials are mixed together in amounts such that there are substantially equal quantities of vanadium in the first and second starting materials. A predetermined volume of a sulphuric acid solution having a predetermined molar concentration is added to produce a vanadyl sulphate ($VOSO_4$) solution having the specified molar concentration.

PROCESS

BACKGROUND TO THE INVENTION

THIS invention relates to a process for the preparation of a vanadyl sulphate solution with a specified molar concentration.

It is known to produce vanadyl sulphate by dissolving vanadium pentoxide in hot dilute sulphuric acid under vigorous agitation and continued heating with the aid of sulphur dioxide as a reducing agent.

The limited solubility of sulphur dioxide in acidic and aqueous solutions results in the emission of sulphur dioxide from the solution and this presents an environmental hazard. Overdosing of the solution with SO_2 gas results in the unwanted formation of the lower valent vanadium sulphate, namely V_2SO_4 and not vanadyl sulphate ($VOSO_4$).

Since the dissolution of vanadium pentoxide in sulphuric acid is endothermic heat has to be provided to drive the formation of vanadyl sulphate.

There is always a need for a new method for the production of vanadyl sulphate.

SUMMARY OF THE INVENTION

According to the invention there is provided a process for the preparation of a vanadyl sulphate solution with a specified molar concentration which includes the steps of:

(1) providing a first starting material containing vanadium pentoxide (V_2O_5);

(2) providing a second starting material containing vanadium trioxide (V_2O_3);

(3) mixing the first and second starting materials in amounts such that there are substantially equal quantities of vanadium in the first and second starting materials; and

(4) adding a predetermined volume of a sulphuric acid solution having a predetermined molar concentration to produce a vanadyl sulphate ($VOSO_4$) solution having the specified molar concentration.

The vanadyl sulphate solution produced may have any desired molar concentration, for example 2M, 4M or up to a maximum of 6M.

The first starting material is preferably substantially pure bulk commercial grade vanadium pentoxide having about a 99,5% vanadium pentoxide content.

The second starting material is generally a commercial grade V_2O_3 powder having an equivalent V_2O_5 content of 118 to 122%.

The sulphuric acid solution preferably has a molarity of from 2,8 to 8,5 depending upon the specified molar concentration of the vanadyl sulphate solution.

Step (4) of the process is exothermic. However for low molar concentrations of, for example <3M vanadyl sulphate, it is preferable to supply heat at the beginning of the dissolution step to trigger the reaction. The heat may be

supplied by adding heated, preferably boiling water to the reaction, or by warming the sulphuric acid solution, e.g to a temperature of about 50°C.

BRIEF DESCRIPTION OF THE DRAWING

Figure 1 is a graph indicating the optimum ratio of V_2O_3, to V_2O_5 needed for a complete reaction, expressed in mVolt.

DESCRIPTION OF EMBODIMENTS

The crux of the invention is a process for the preparation of a vanadyl sulphate solution with a specified molar concentration, from a first starting material containing V_2O_5 and a second starting material containing V_2O_3, and a sulphuric acid solution.

The reaction proceeds according to the following formula:

$$V_2O_3 + V_2O_5 + 4H_2SO_4 \rightarrow 4VOSO_4 + 4H_2O.$$

The reaction may be monitored by measuring the reduction potential, as is illustrated in Figure 1. The start of the production of vanadyl sulphate is illustrated at the point where the graph dips sharply.

Although the reaction between the V_2O_5, V_2O_3 and a sulphuric acid solution is exothermic, for low molar concentrations of, for example <3M vanadyl sulphate, it is preferable to add heat at the beginning of the dissolution step to trigger the reaction. The heat may be added in the form of heated, preferably boiling water, or by heating the sulphuric acid solution to a temperature of approximate 50°C.

The advantages of the process of the invention are firstly that various specified molar concentrations of vanadyl sulphate can be produced, secondly that no additional chemicals are required for the reaction, and thirdly that the process is environmentally friendly.

Examples

Various examples of the invention will now be given.

The following mixtures were used for the preparation of 2, 4 and 6 molar solutions of vanadyl sulphate:

Hivox (V_2O_3 powder) quality – 119,6% expressed as percent V_2O_5

Molarity	V_2O_5	Hivox	50% H2SO4	Boiling Water
2	4,5g	4,1g	15ml	35ml
4	9,0g	8,3g	30ml	20ml
6	13,5g	12,5g	45ml	5ml

The dissolution time was 15 minutes.

The 2 and 4 molar solutions were filtered whilst warm using a laboratory vacuum pump with a GF/C fiber filter. It was not possible to filter the 6 molar solution because of its viscosity.

CLAIMS

1. A process for the preparation of a vanadyl sulphate solution with a specified molar concentration includes the steps of:
   (1) providing a first starting material containing vanadium pentoxide (V_2O_5);
   (2) providing a second starting material containing vanadium trioxide (V_2O_3);
   (3) mixing the first and second starting materials in amounts such that there are substantially equal quantities of vanadium in the first and second starting materials; and
   (4) adding a predetermined volume of a sulphuric acid solution having a predetermined molar concentration to produce a vanadyl sulphate ($VOSO_4$) solution having the specified molar concentration.

2. A process according to claim 1, wherein the vanadyl sulphate solution produced has a molar concentration up to a maximum of 6M.

3. A process according to claim 1 or claim 2, wherein the first starting material is substantially pure bulk commercial grade vanadium pentoxide having about a 99,5% vanadium pentoxide content.

4. A process according to any one of the preceding claims wherein the second starting material is a commercial grade V_2O_3 powder having an equivalent V_2O_5 content of 118 to 122%.

5. A process according to any one of the preceding claims wherein the sulphuric acid solution has a molarity of from 2,8 to 8,5 depending upon the specified molar concentration of the vanadyl sulphate solution.

6. A process according to any one of the preceding claims, wherein at low molar concentrations of the vandyl sulphate produced in step 4) heat is supplied at the beginning of step 4) to trigger the reaction.

7. A process according to claim 6, wherein the heat is supplied by adding heated water of by warming the suphuric acid solution.

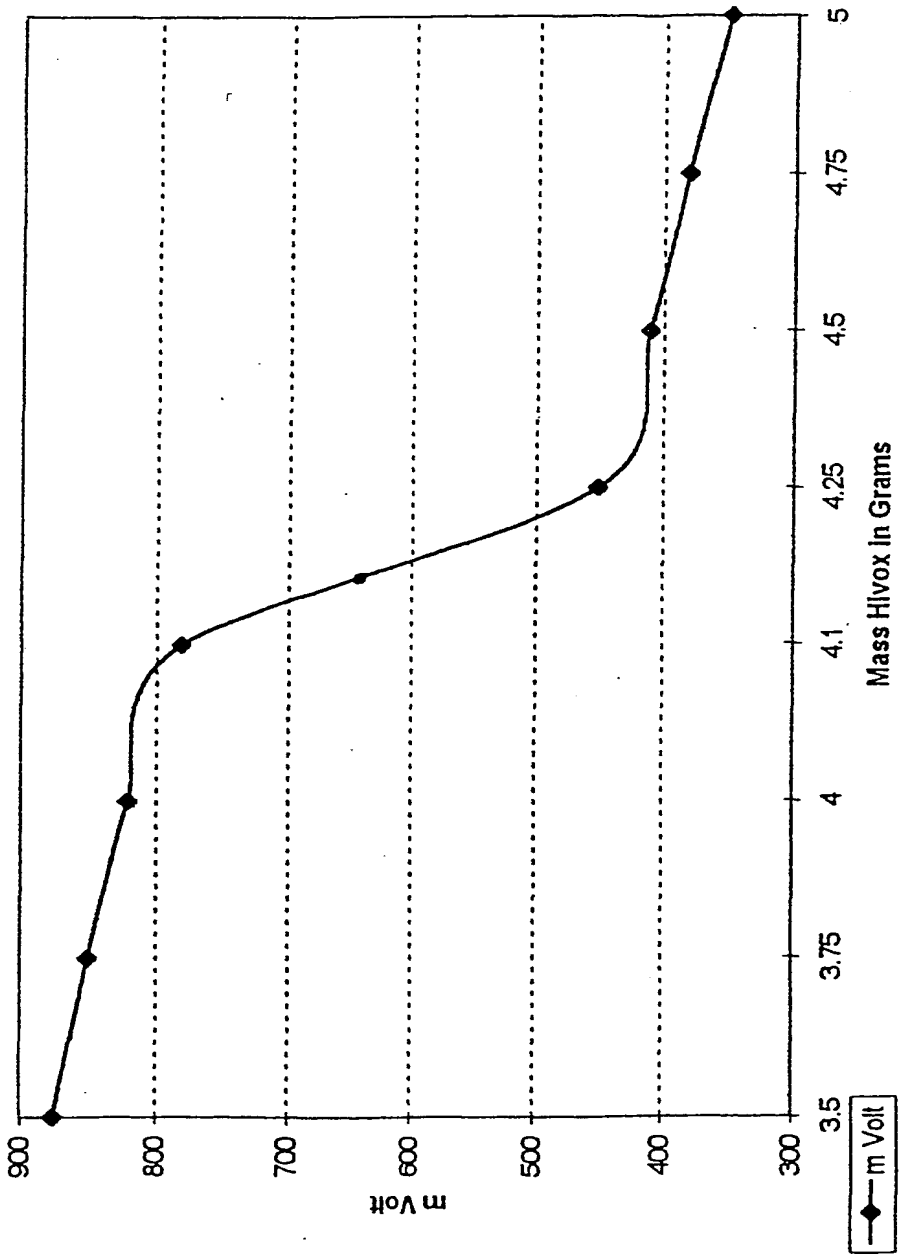
m Volt
900
800
700
600
500
400
300
3.5
3.75
4
4.1
4.25
4.5
4.75
5
Mass Hivox In Grams
m Volt

Figure 1

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(54) Title: METHOD FOR PREPARING VANADYLSULFATE

(57) Abstract: A process for the preparation of a vanadyl sulphate solution with a specified molar concentration from a first starting material containing vanadium pentoxide (V_2O_5) and a second starting material containing vanadium trioxide (V_2O_3). The first and second starting materials are mixed together in amounts such that there are substantially equal quantities of vanadium in the first and second starting materials. A predetermined volume of a sulphuric acid solution having a predetermined molar concentration is added to produce a vanadyl sulphate ($VOSO_4$) solution having the specified molar concentration.

INTERNATIONAL SEARCH REPORT

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A. CLASSIFICATION OF SUBJECT MATTER
IPC 7 C01G31/00

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 7 C01G

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)
EPO-Internal, WPI Data, PAJ, CHEM ABS Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	PATENT ABSTRACTS OF JAPAN vol. 2000, no. 06, 22 September 2000 (2000-09-22) & JP 2000 072441 A (TAIYO KOKO CO LTD), 7 March 2000 (2000-03-07) abstract	1
A	DD 151 923 A (LADWIG GERHARD;OLIEW ELKE) 11 November 1981 (1981-11-11) claim 1	1

☐ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

° Special categories of cited documents :

"A" document defining the general state of the art which is not considered to be of particular relevance

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"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
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Name and mailing address of the ISA	Authorized officer
European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Tx. 31 651 epo nl, Fax: (+31-70) 340-3016	Siebel, E

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INTERNATIONAL SEARCH REPORT

Information on patent family members

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Patent document cited in search report	Publication date	Patent family member(s)	Publication date
JP 2000072441 A	07-03-2000	NONE	
DD 151923 A	11-11-1981	DD 151923 A1	11-11-1981